Environment Canada Environnement Canada
0050393J DEST PVB EXCH COPY 03
TP 360 E5613
VOL ISS 257
ENFOR PROJECT C

ENFOR Project C-257

Effect of Particle Size on Gross Heat of Combustion of Wood

by

John M. Kryla

Contractor's Final Report 1/
DSS Contract 43SS.KN107-1-4318

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March 1983

ACKNOWLEDGEMENTS

The author wishes to thank Dr. R.P. Overend, National Research Council. for his constructive suggestions during the course of this project. The assistance of Miss B.S. Grant and Mr. D.R. Huffman, FORINTEK Canada Corp., is appreciated.

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ABSTRACT

Three separate experiments were conducted to examine the effects of (1) particle size, (2) drying method, and (3) radial position in stem on the calorific value of wood. In the particle size experiment, wood samples of kiln-dried trembling aspen (*Populus tremuloides* Michx.) and white spruce (*Picea glauca* (Moench) Voss) were comminuted in a laboratory Wiley mill and separated to yield 6 particle size classes (2,000-3,350, 250-425, 150-250, 75-150, 38-75, and <38 μm). The samples were then ovendried before testing. In the drying study, freshly cut trembling aspen and white spruce samples were comminuted to a coarse fraction (2,000-3,350 μm) and a fine fraction (<250 μm). The two particle size classes were then subjected to either air-drying or ovendrying prior to further analyses. To examine radial variability, earlywood and latewood of various growth rings and inner and outer bark were removed from a 10-year old stem section of hybrid poplar (*Populus euramericana* (Dode) Guinier, clone I-262). The hybrid poplar materials were comminuted to less than 250 μm and ovendried prior to testing. All samples were finally stored over a saturated solution of potassium carbonate to achieve an equilibrium moisture content of 6 to 7 percent (wet basis) prior to oxygen bomb testing.

Calorific values of all materials were determined utilizing adiabatic oxygen bomb calorimetry. Lignin, holocellulose, α-cellulose, and extractive contents of the aspen, spruce and hybrid poplar were determined using standard techniques. Selected samples were analyzed

for carbon, hydrogen, nitrogen, and ash concentrations. Experimental calorific values (adjusted to a dry basis) were compared to theoretical values calculated from the constituent and elemental analysis data.

Analysis of variance indicated that particle size, drying method, and radial position had small but significant effects upon the measured wood calorific value (possible variation of up to about 4.3%). To minimize variation, it is recommended that a standardized practice be utilized in the preparation of wood for calorific analyses.

1. INTRODUCTION

Standard methods of wood analyses frequently require that the material be reduced (comminuted) to a specified size range prior to testing. For most chemical analyses, the fines are discarded prior to testing even though, due to fractionation, the retained sample may then not be totally representative of the original material (Harris 1953, Merrill and French 1964, Browning 1967, Ottone and Baldwin 1981).

It is recommended that a particle size smaller than 250 μm be utilized in determinations of gross heat of combustion by oxygen bomb calorimetry (Anon. 1975). Wood and bark calorific values have been determined using various particle sizes, usually without exclusion of the fine materials (Howard 1973, Murphey and Cutter 1974, Bowersox et al 1979, Sastry and Anderson 1980). Where correlations of heating value to chemical characteristics are desired, the materials are often sieved to a specified size range prior to oxygen bomb calorimetry (Murphey and Masters 1978, Sandala et al 1981). In most cases, the materials are ovendried before calorific testing.

Recent research conducted at the University of Toronto has indicated that a decrease in particle size will achieve an increase in the gross heat of combustion of wood (Sastry 1981). Comminuted juvenile hybrid poplar (2-year-growth) wood and/or bark, less than 75 μm in size, yielded heating values ranging from about 8 to 20 percent higher than the values from corresponding particles larger than 850 μm in size. These results prompted discussions concerning the potential energy benefits in comminuting wood to a fine particle size. However, since these initial tests were conducted on physically and chemically heterogeneous materials which may have been subject to substantial fractionation during grinding, additional research was necessary to fully evaluate the effects of particle size and other possible factors on the calorific value of wood.

The major objective of this study is: to examine the calorific value of wood, as affected by:

(a) particle size

(b) drying method

(c) radial position in stem

2. METHODS AND MATERIALS

2.1 Particle Size Experiment

2.1.1 Material Preparation for Chemical Analyses

Material for the particle size experiment was prepared from flat-sawn, kiln-dried trembling aspen (*Populus tremuloides* Michx.) and white spruce (*Picea glauca*(Moench) Voss) lumber. Selection of material was limited to defect-free sapwood portions of the lumber. The average growth rates of the aspen and spruce were 3.5 and 4 annual rings/cm, respectively. Specific gravity (ovendry mass/ovendry volume) was 0.50 for the aspen and 0.41 for the spruce.

The base material ("non-comminuted") for all tests was prepared by reducing the wood to a size ranging from 2,000 to 3,350μm in a modified hammermill. For chemical analyses, the base material was ground in a Thomas Wiley mill (model A75-A, 1,725 rpm) (Fig. 1)fitted with a 425 μm sieve. The ground material was then sieved to a 180-250 μm particle size test fraction utilizing a Ro-Tap shaker.

Lignin contents of the spruce and aspen were determined in duplicate on ovendried, extractive-free material using standard procedures (ASTM 1981b, c). Holocellulose was prepared in duplicate from ovendried wood using the acid chlorite method (Browning 1967). One holocellulose sample

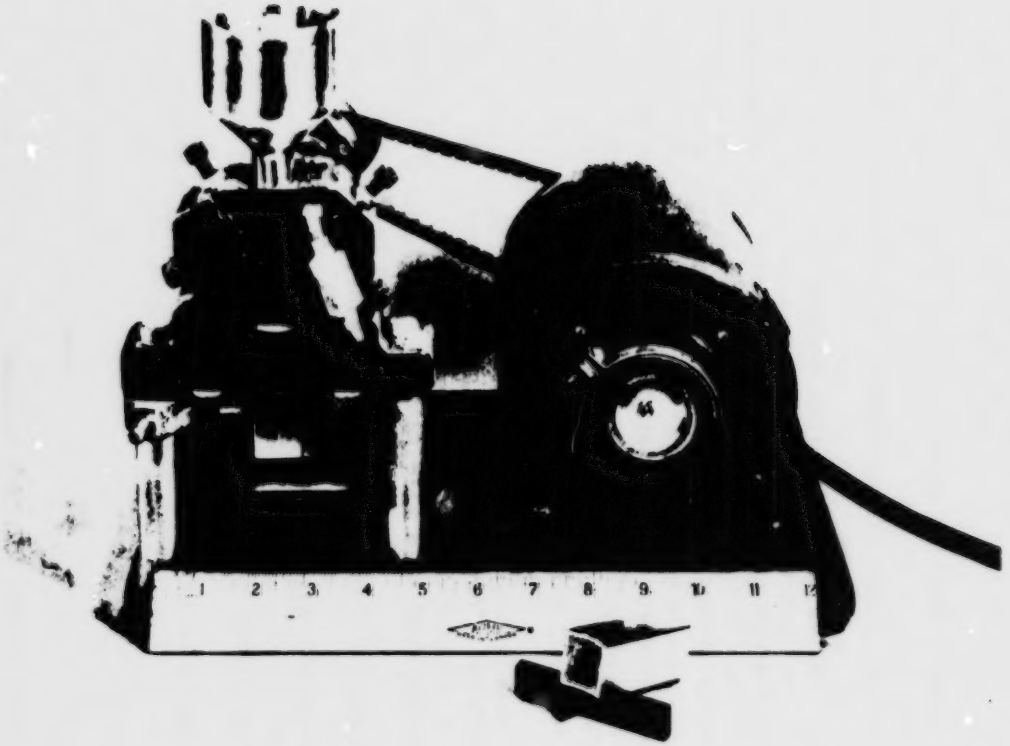

Figure 1. Thomas Wiley mill utilized for comminution.

was ovendried and corrected for residual lignin while the other was air-dried and used for determination of α-cellulose. Alpha-cellulose content was determined in duplicate according to recommended techniques (ASTM 1981a). Constituent concentrations were expressed as a percentage of the initial ovendry, unextracted wood mass.

Sample filtering during preparation of extractive-free wood resulted in some loss of materials, prohibiting an accurate determination of extractive content. For evaluation of extractive content, additional extractions were conducted on both 2,000-3,350 μm and 180-250 μm particle sizes of spruce and aspen without removal of the material from the Soxhlet apparatus.

Carbon, hydrogen, and nitrogen levels of the spruce and aspen were determined using a Perkin Elmer model 240B elemental analyzer following recommended procedures (Anon. 1980). Tests were conducted in triplicate on ovendry materials of 2 particle sizes (250-425 μm and 38-75 μm). Ash contents, conducted in triplicate, were determined by placing crucibles containing 1.5 g ovendry samples in a muffle furnace at about 600°C for 7 hours. Since sulfur was assumed to be negligible, oxygen was determined by difference. Three elemental analyses tests were also conducted on extractive-free 180-250 μm spruce.

2.1.2 Material Preparation for Calorific Analyses

2.1.2.1 Standard and Batch Comminution

Two methods of comminution were utilized for preparation of 6 particle sizes of spruce and aspen wood: (1) the "standard" method, and (2) the "batch" process. By the standard technique, the base 2,000-3,350 μm material was ground in a Wiley mill fitted with a 425 μm sieve. The comminution time was found to be approximately 40 seconds per g of wood. In the batch method, the exit screen was sealed and about 1 g of 2,000-3,350 μm wood was placed between the knives; comminution was then conducted for only 10 seconds. The batch method was utilized to minimize potential thermal effects and simulate "immediate" cell wall fractionation.

During initial grinding, the temperature of the cutting chamber was monitored by a suitably located thermocouple. During several standard runs, the mill was also sealed to allow sampling of gases which may have been produced during comminution.

After comminution, the particles were classified using a Ro-Tap sieve shaker into particle size classes of 250-425, 150-250, 75-150, 38-75, and less than 38 μm. The original base 2,000-3,350 μm wood particles were included in the testing plan to represent coarse, non-comminuted material.

2.1.2.2 Extended Duration Comminution

Extended duration comminution was conducted to reduce the entire original sample to a uniform particle size, thus minimizing cell wall fractionation effects. By this method, the spruce and aspen were ground for about 30 minutes each in the Wiley mill with the exit screen sealed. The final test samples consisted primarily of materials smaller than 150 μm which were classified into particle sizes of 75-150 and 38-75 μm. Materials less than 38 μm were not utilized.

2.1.3 Adiabatic Oxygen Bomb Calorimetry

For adiabatic oxygen bomb calorimetry, samples of approximately 0.5 g were ovendried (in a forced-air oven at 103°C for 24 hours), weighed and stored over a saturated salt solution of potassium carbonate until a constant mass was achieved. (It is recommended (Anon. 1975) that some moisture be present in the sample to reduce the combustion rate and to retain the particles in the capsule.) Equilibrium moisture content (emc) over the salt solution was approximately 6.8 percent (wet basis) for the spruce and 5.9 percent for aspen. No effect of particle size upon emc was discernible.

Calorific values were deterimined using a Parr model 1241 adiabatic oxygen bomb calorimeter at 3.0 MPa oxygen pressure following prescribed methods (Anon. 1975, ASTM 1977), without nitric and sulfuric acid titrations. Combustion was considered complete when the oxygen bomb contained no residue left visible to the naked eye. Calorific values were deterimed in triplicate and expressed on a dry basis. For the materials produced by

standard and batch comminution, a total of 72 oxygen bomb tests were conducted using loosely packed samples (2 species x 2 grinding methods x 6 particle sizes x 3 replications). For the samples prepared by extended duration comminution, calorific tests were conducted on both loosely packed and pelletized materials. For these materials, a total of 24 oxygen bomb tests were conducted (2 species x 2 particle sizes x 2 methods of packing x 3 replications).

In addition, 3 oxygen bomb tests each were conducted on extractive-free 2,000-3,350 and 180-250 μm spruce and aspen.

Reference oxygen bomb tests were conducted on 5 particle sizes of sucrose. An aqueous sucrose solution was crystallized on a shallow dish. After drying, the material was comminuted using a mortar and pestle to particle sizes of 2,000-4,750, 250-425, 75-250, 38-75, and less than 38 μm. Three oxygen bomb tests were conducted for each of the particle sizes.

Experimentally determined wood calorific data were compared to theoretical values calculated using the Institute of Gas Technology (IGT) correlation equation (Graboski and Bain 1979) described as follows:

$$CV = [146.58\ C + 568.78\ H + 29.45 - 6.58\ A - 51.53\ (O + N)]/429.917 \quad (1)$$

where

CV = calorific value, MJ/kg

A = ash, C = carbon, H = hydrogen, N = nitrogen, O = oxygen, expressed as percentage of original ovendry mass.

2.2 Drying Experiment

Sapwood portions of freshly cut white spruce and trembling aspen were selected for the drying study. The average growth rates of the spruce and aspen were 4.7 and 4 annual rings/cm, respectively. The materials were sliced into strips and partially air-dried prior to reduction into 2 particle size classes. A coarse segment was produced by manually cutting the strips into particles (non-comminuted) ranging from 2,000-3,350 μm. This method of particle reduction was chosen to minimize potential volatile loss and other thermal alterations due to grinding. A fine fraction was produced by comminuting the samples in a Wiley mill to pass a 250 μm sieve. The 2 particle sizes of spruce and aspen were subjected to 2 different methods of drying. In the first method, samples of approximately 0.5 g were ovendried, weighed, and stored over a saturated salt solution of potassium carbonate prior to oxygen bomb calorimetry. In the second method, materials were stored over the salt solution without ovendrying.

Calorific values were determined in triplicate by the previously described methods and expressed on a dry basis. For the materials which had not been ovendried, representative moisture content samples were removed prior to oxygen bomb testing. Emc's of the various samples prior to oxygen bomb calorimetry are summarized in Table 9.

2.3 Radial Position Experiment

A stem section of 10-year-old hybrid poplar (*Populus euramericana* (Dode) Guinier, clone I-262) was supplied to Forintek by the Ontario Ministry of Natural Resources, Maple, Ontario (Fig. 2). The material was cut in January, 1982, and stored frozen until required for experimentation. The stem section measured approximately 30 cm in both diameter and length. The average growth ring width was 14 mm with minimum and maximum ring widths of 5 mm (ring 1, from pith) and 20 mm (rings 3 and 6), respectively. Inner bark thickness ranged from 5-7 mm while outer bark varied from 0-7 mm.

Material for analyses was prepared by removing a test section which measured approximately 5 and 25 cm in the tangential and longitudinal directions, respectively, and contained all annual rings on one side of the pith (Fig. 2).

Lignin, holocellulose, α-cellulose, and extractive contents were determined by the previously described methods on materials from ring 4 and ring 9. Although the entire section may have consisted of juvenile wood, the material from ring 4 was classified as heartwood while that of ring 9 was termed sapwood.

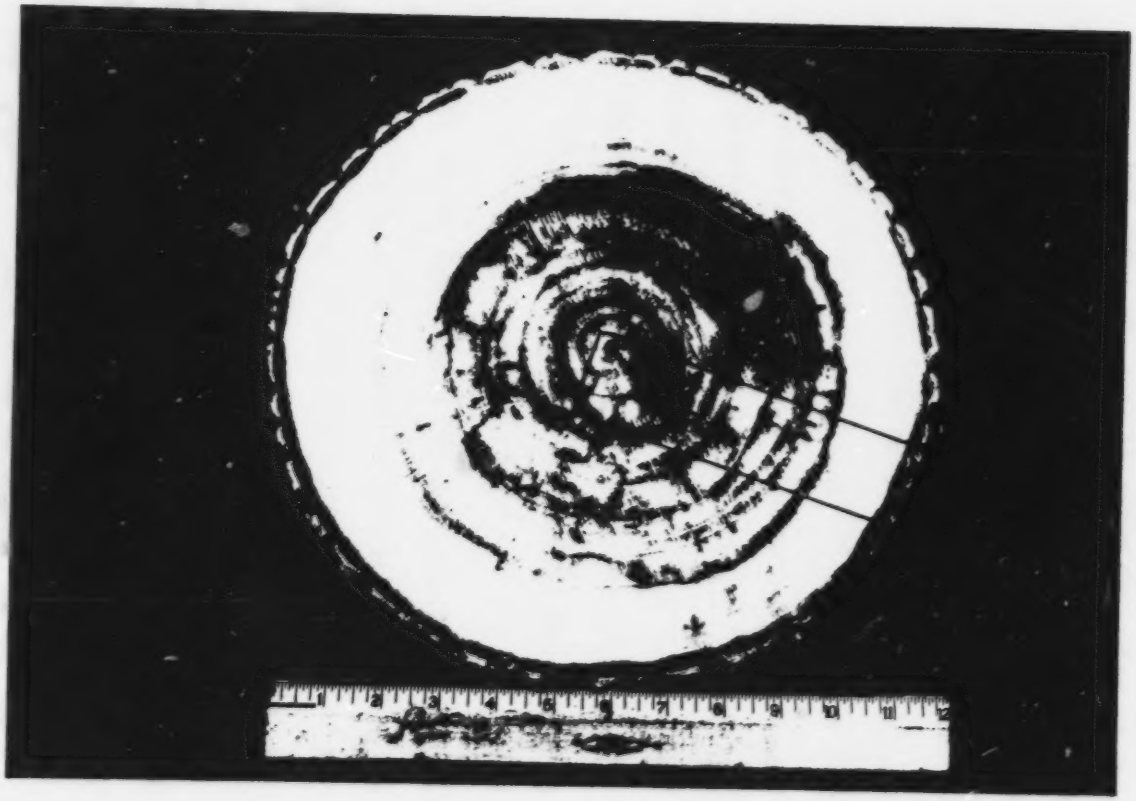

Figure 2. Hybrid poplar clone I-262 cross-section (rectangular area represents test section).

Ring numbers 1,3,5,7 and 10 and inner and outer bark components were removed from the test section for calorific and elemental analyses. Rings 3,5,7 and 10 were divided into earlywood and latewood samples by selecting material from the inner and outer 3 mm of each growth ring. For ring 1, a mixture of earlywood and latewood was utilized. All materials were comminuted to pass a 250 μm sieve and ovendried prior to analyses. Samples were stored over a saturated solution of potassium carbonate to achieve an emc of approximately 6 percent prior to oxygen bomb calorimetry. Calorific value, carbon, hydrogen, and nitrogen were determined in triplicate by the previously described methods. Ash content was conducted in duplicate. The elemental analysis data were used in equation [1] to calculate theoretical calorific values of the various hybrid poplar components.

Specific gravity (ovendry mass/ovendry volume) was determined in triplicate for the earlywood and latewood portions of rings 3,5,7 and 10.

3. RESULTS AND DISCUSSION

3.1 Particle Size Experiment

3.1.1 Constituent and Elemental Analyses

Lignin content of the aspen was found to be 17.6 percent, compared to 26.8 percent for spruce (Table 1). Holocellulose content, corrected for residual lignin, was 78.7 percent for aspen and 69.2 percent for spruce. Summations of lignin, holocellulose, and extractive contents yielded totals of 100.0 and 98.5 percent for aspen and spruce, respectively.

Elemental analysis data for the 250-425 μm and 38-75 μm particle size classes of aspen and spruce are outlined in Table 2. Analysis of variance (Tables A1-A3) did not show differences in elemental composition due to particle size. Average carbon, hydrogen, nitrogen, and ash levels for aspen were 48.84, 6.13, 0.10, and 0.16 percent, respectively. Respective levels for white spruce were 49.65, 6.13, 0.10 and 0.20 percent. Carbon and hydrogen contents of extractive-free white spruce were lower than for unextracted samples.

3.1.2 Particle Size Distribution after Standard and Batch Comminution

Particle size distribution after standard and batch comminution is summarized in Table 3. Due to the longer residence time, standard comminution generated more material smaller than 250 μm

Table 1. Constituent and extractive contents of trembling aspen and white spruce*

Species	Lignin	Holocellulose	α-Cellulose	Extractives
	----------% ----------			
Trembling aspen	17.6	78.7	46.4	3.7
White spruce	26.8	69.2	45.0	2.5

*Values based upon ovendry unextracted wood. Lignin and α-cellulose values are average of 2 observations while holocellulose and extractive values are based on 1 observation.

Table 2. Elemental analysis of 250-425 and 38-75 μm trembling aspen and white spruce and extractive-free 180-250 μm white spruce*

Species	Particle size, μm	Carbon	Hydrogen	Nitrogen	Oxygen	Ash
		%				
Trembling aspen	250-425	48.97	6.19	0.08	44.56	0.20
	38-75	48.72	6.07	0.13	44.96	0.12
White spruce	250-425	49.70	6.13	0.03	43.94	0.20
	38-75	49.59	6.12	0.00	44.08	0.21
Extractive-free white spruce	180-250	49.49	5.94	0.34	44.06	0.17

*Values based upon ovendry wood. Values are average of 3 observations; oxygen determined by difference.

Table 3. Particle size distribution of trembling aspen and white spruce after standard and batch comminution*

Particle size, μm	Trembling aspen		White spruce	
	Standard comminution	Batch comminution	Standard comminution	Batch comminution
	% particle size distribution			
2,000-3,350	-	17.4	-	16.7
425-2,000	12.9	34.9	5.6	37.3
250-425	40.6	27.9	36.1	23.3
150-250	9.0	10.1	27.5	11.2
75-150	33.4	6.8	22.3	7.6
38-75	2.4	2.1	6.5	2.6
< 38	1.7	0.8	2.0	1.4

*Computations based on air-dry weight.

in size than did the batch process. (Grinding with re-sharpened mill blades during the latter segment of the study was found to produce substantially more fine materials). Due to the short duration of the batch process, a substantial amount of material in the 2,000-3,350 μm class remained after comminution.

Although fitted with a 425 μm sieve, some material coarser than 425 μm remained after standard comminution. The placement of the thermocouple wire under the protective cover of the mill chamber caused some material loss before complete grinding. These materials were not utilized in testing. After batch comminution, approximately 35 percent of the aspen and 37 percent of the spruce were in the 425-2,000 μm particle size class and not used in calorific testing.

During standard comminution, the temperature of the mill chamber reached a maximum of about 75°C; the actual temperature of the wood particle surfaces, however, may have been significantly higher. During batch comminution, the mill temperature did not increase above the ambient level.

Gas analysis during standard comminution of spruce did not show an increase in carbon dioxide content. Significant degradation of wood constituents during comminution would have presumably caused a detectable increase in carbon dioxide level.

3.1.3 Calorific Values after Standard and Batch Comminution

Calorific values of the aspen and spruce wood produced by standard and batch comminution are summarized in Tables 4 and 5 and shown graphically in Figures 3 and 4. Analysis of variance (Table A4) showed that particle size had a significant effect upon the calorific values of both species. Comparison of mean values by Duncan's multiple range test (Steel and Torrie 1960) showed that the highest heating values were generally measured for the coarse (non-comminuted 2,000-3,350 μm) and fine (38-75 and <38 μm) materials. The intermediate particle size classes (250-425, 150-250, and 75-150 μm) exhibited the lowest calorific values. These variations in calorific values may have resulted from differences in chemical composition due to cell wall fractionation after comminution. Harris (1953) observed that during grinding

Table 4. Calorific values of trembling aspen as affected by particle size and method of comminution*

Particle size, µm	Calorific value (MJ/kg)			
	Standard comminution		Batch comminution	
	Average	Range	Average	Range
2,000-3,350	19.56a	19.40-19.68	19.50ab	19.36-19.74
250-425	19.01e	18.90-19.10	19.31bcd	19.19-19.37
150-250	19.23cd	19.13-19.30	19.23cd	19.19-19.29
75-150	19.24cd	19.15-19.31	19.29cd	19.14-19.43
38-75	19.19de	19.11-19.29	19.64a	19.60-19.69
<38	19.52ab	19.41-19.65	19.42abc	19.37-19.52

*Values based upon ovendry wood; averages are based upon 3 observations. Mean values with the same letter are not significantly different at the 5 percent level.

Table 5. Calorific values of white spruce as affected by particle size and method of comminution*

Particle size, μm	Calorific value (MJ/kg)			
	Standard comminution		Batch comminution	
	Average	Range	Average	Range
2,000-3,350	20.01ab	19.94-20.06	20.14a	20.05-20.31
250-425	19.70cd	19.51-19.80	19.60de	19.48-19.74
150-250	19.45e	19.35-19.57	19.69cd	19.60-19.84
75-150	19.86bc	19.84-19.88	19.60de	19.56-19.64
38-75	19.87bc	19.72-19.98	19.81bcd	19.74-19.94
<38	19.93ab	19.82-20.05	19.70cd	19.67-19.73

*Values based upon ovendry wood; averages are based upon 3 observations. Mean values with the same letter are not significantly different at the 5 percent level.

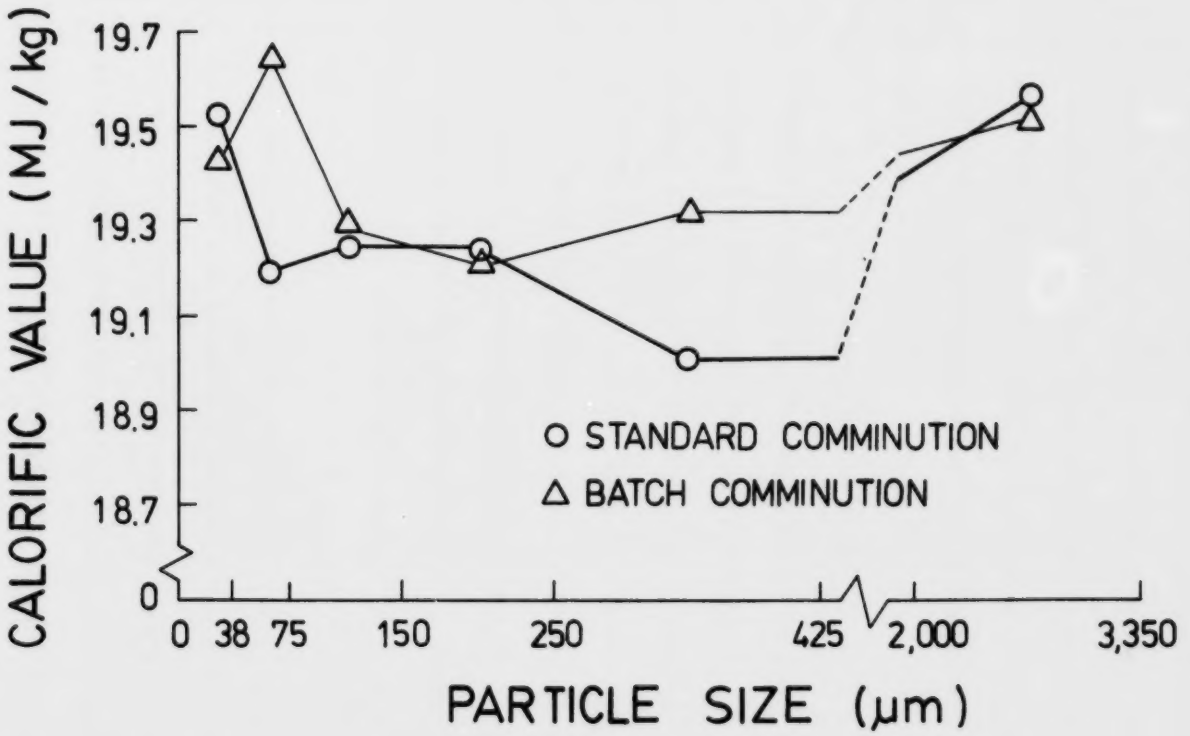

Figure 3. Relationship between calorific value, particle size, and method of comminution for trembling aspen (particle size is plotted as an average value; note change in scale).

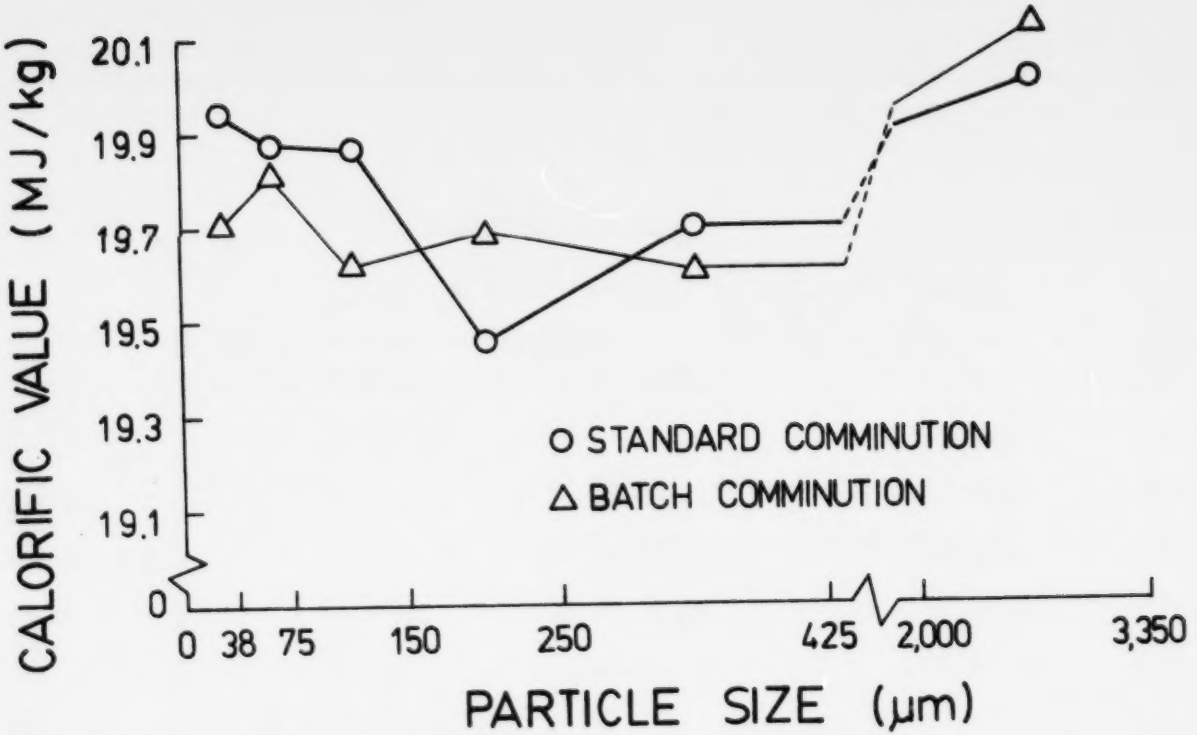

Figure 4. Relationship between calorific value, particle size, and method of comminution for white spruce (particle size is plotted as an average value; note change in scale).

of white ash, the lignin rich middle lamella portion tended to generate more fine materials than did the other components of the cell wall. Since lignin has the highest heating value of the wood constituents, fines from a sieved sample would presumably have a higher calorific value than the larger particles. Grinding of the base material may also have expelled volatile extractives, as indicated by the lower calorific values of the intermediate particle size classes.

Although significant, the differences between average maximum and minimum calorific values were less than 3 percent for both aspen and spruce. Larger variations may be encountered for species such as ash and oak which have a less uniform cellular structure than spruce and aspen. Contrary to the findings of the University of Toronto, this present work has found no evidence of a linear increase in wood calorific value with a decrease in particle size.

For comparison, calorific values of the 5 reference sucrose particle sizes are shown in Table 6. Analysis of variance (Table A5) did not show a significant treatment effect, indicating that for chemically homogeneous substances, particle size has little influence upon gross heat of combustion.

Table 6. Average and range of sucrose calorific values*

Particle size, μm	Calorific value (MJ/kg) Average	Range
2,000-4,750	16.34	16.32-16.37
250-425	16.49	16.45-16.54
75-250	16.47	16.42-16.52
38-75	16.37	16.24-16.45
< 38	16.45	16.38-16.55

* Average values are based upon 3 observations.

The grinding method in itself did not significantly affect wood calorific value. However, as indicated by the interactions in Table A4, some variations were observed between samples produced by the 2 different methods of comminution. As mentioned previously, the particle size distribution after standard comminution was substantially different than that after batch comminution. Furthermore, a high percentage of the batch material (425-2,000 μm materials, Table 3) was not utilized in calorimetry. It is thus possible that similar particle size classes produced by the 2 methods of grinding may have differed in chemical composition.

The mean calorific value of spruce was 19.80 MJ/kg, compared to 19.29 MJ/kg for aspen. The higher calorific value of spruce can be attributed to larger amounts of lignin (Table 1). Murphey and Masters (1978) found that isolated α-cellulose, hemicellulose, and lignin from red oak had calorific values of 17.03, 16.67 and 21.18 MJ/kg, respectively. When the fractional constituent concentrations were multiplied by their individual calorific values, the theoretical wood calorific value was calculated to be 10.28 percent lower than the experimental value. The authors stated that, due to degradation, the heating values of the isolated constituents may be lower than those in the native form.

When adjusted to 100 percent, the lignin, α-cellulose, hemicellulose, and extractive contents of the trembling aspen examined in this study are 17.62, 46.44, 32.20 and 3.74 percent, respectively (hemicellulose calculated as the difference between holocellulose and α-cellulose contents). For the spruce, respective values are 27.18, 45.67, 24.57 and 2.58 percent. Using the constitutent heating value data of Murphey and Masters (1978) and an average extractive calorific value equal to that of the lignin, the theoretical calorific value of aspen is calculated to be 17.80 MJ/kg, or 7.73 percent lower than the experimental value. For spruce, the theoretical heating value is calculated to be 18.18 MJ/kg, or 8.20 percent lower than the experimental.

The utilization of the IGT equation and the average elemental analysis data of Table 2 was found to give a closer approximation of calorific value than did correlation to constituent concentration. As shown in Table 7, theoretical heating values calculated using the IGT equation were, with one exception, within 1 percent of the experimental values.

Table 7. Comparison of experimental to theoretical calorific values of trembling aspen and white spruce*

Species	Particle size, μm	Calorific value (MJ/kg)		Error %
		Experimental	Theoretical according to IGT equation [1]	
Unextracted				
Trembling aspen	2,000-3,350	19.56	-	-
	250-425	19.01	19.60	+3.09
	150-250	19.23	-	-
	38-75	19.19	19.30	+0.59
White spruce	2,000-3,350	20.01	-	-
	250-425	19.70	19.85	+0.91
	150-250	19.45	-	-
	38-75	19.87	19.79	-0.37
Extractive-free				
Trembling aspen	2,000-3,350	19.19	-	-
	180-250	18.57	-	-
White spruce	2,000-3,350	19.80	-	-
	180-250	19.40	19.47	+0.38

*Values based upon ovendry wood. Experimental values are based upon 3 observations.

3.1.4 Calorific Values after Extended Duration Comminution

Extended duration comminution did not cause noticeable alterations in the appearance of the wood particles. Particle size appeared to reach a minimum within a relatively short time period and further comminution was ineffective in reducing particle dimensions.

Calorific values of the 75-150 and 38-75 μm spruce and aspen produced by extended duration comminution were generally comparable to the data obtained from the standard and batch comminution tests (Table 8). Pelletization of the 75-150 and 38-75 μm spruce was found to result in calorific values which were about 2 to 3 percent higher than those of the respective loosely packed samples, although inspection of bomb contents did not indicate differences in combustion behaviour. On the other hand, calorific value of the 38-75 μm pelletized aspen was similar to that of the loosely packed 38-75 μm material. Even though several tests were attempted, 75-150 μm aspen materials which were pelletized did not undergo complete combustion, whereas loosely packed 75-150 μm samples exhibited complete combustion.

It is suggested (Anon. 1975) that pelletization of fine particles may be required to prevent ejection of material from the sample cup during combustion. In the present work,

Table 8. Calorific values of trembling aspen and white spruce after extended duration comminution to final mesh sizes of 75-150 and 38-75 μm*

Sample form for oxygen bomb test	Calorific value (MJ/kg)			
	Trembling aspen		White spruce	
	75-150 μm	38-75 μm	75-150 μm	38-75 μm
Loosely packed	19.16	19.41	19.53	19.49
Pelletized	**	19.40	20.05	20.09

*Values based upon ovendry wood and are average of 3 observations.

**Pelletized 75-150 μm trembling aspen exhibited incompelte combustion in all tests.

there was no difficulty in obtaining complete combustion of loosely packed, fine particles. Similarly, extensive research (Murphey and Cutter 1974, Murphey and Masters 1978, Bowersox et al 1979) has been conducted on (apparently) non-pelletized wood materials without combustion problems. Howard (1973) however, reported that a small amount of carbon consistently remained after combustion of various southern pine materials. In the worst case, the residual carbon contributed to a variation of about 0.12 MJ in calorific value. Musselman and Hocker (1981) conducted tests on comminuted (unspecified particle size), pelletized wood and bark samples without evidence of incomplete combustion. On the other hand, Neenan and Steinbeck (1979) noted that pelletized samples consisting of particles smaller than 850 μm were not successfully combusted during oxygen bomb calorimetry. The authors found that larger particles were more suitable for oxygen bomb calorimetry. It is thus evident that sample pelletization does not necessarily insure complete combustion and should not be considered as a standard preparatory technique for adiabatic oxygen bomb calorimetry.

3.1.5 Calorific Values of Extractive-Free Materials

Calorific values of the extractive-free 2,000-3,350 and 180-250 μm particle size classes of spruce and aspen were approximately 1.5 percent lower than respective unextracted values (Table 7). As observed for the unextracted material, calorific values of extractive-free 180-250 μm materials were approximately 2-3 percent lower than those of the extractive-

free 2,000-3,350 µm specimens. This suggests that the chemical composition of the 180-250 µm particle size classes differed from that of the base 2,000-3,350 µm materials. Since the samples had been extracted prior to oxygen bomb calorimetry, it appears that volatile extractive loss during comminution was not a major factor contributing to the observed particle size variations in calorific value. The majority of volatile loss had presumably occurred during previous kiln-drying of the materials.

3.2 Drying Experiment

Calorific values and emc's of the white spruce and trembling aspen which were dried from the green condition are outlined in Table 9. Material which had not been ovendried achieved a higher emc over the saturated solution of potassium carbonate than did the samples which were ovendried before storage. This corresponds to the typical sorption isotherm which shows that the initial desorption emc of green wood is higher than following adsorption and desorption emc's after ovendrying.

Analysis of variance (Table A6) showed that particle size, species, and method of drying all had significant effects upon calorific value. The calorific value of spruce was higher than that of aspen. As observed in the particle size experiment,

Table 9. Calorific values of freshly cut white spruce and trembling aspen as affected by drying method and comminution*

Species	Calorific Value (MJ/kg)			
	Non-comminuted (particle size = 2,000 - 3,350 μm)		Comminuted (particle size = < 250 μm)	
	Ovendried	Air-dried	Ovendried	Air-dried
White spruce	20.45a (6.82)	20.56a (8.02)	19.68c (6.78)	20.16b (7.04)
Trembling aspen	19.41de (6.03)	19.42de (7.45)	19.32e (6.12)	19.56cd (7.24)

* Values based upon ovendry wood and are average of 3 observations. Values with the same letter are not significantly different at the 5 percent level.

Values in parentheses represent average equilibrium moisture content over saturated solution of potassium carbonate prior to oxygen bomb test.

the average calorific value of the non-comminuted 2,000-3,350 μm material was higher than that of the finely comminuted material. Material which was air-dried had a higher mean heating value than samples which had been ovendried.

Analysis of the significant interactions indicated that the effect of particle size depended upon the species being tested. As shown by Duncan's comparison of means (Table 9), the calorific value of 2,000-3,350 μm spruce was higher than that of the finely ground material. For aspen, particle size by itself did not significantly affect calorific value. For both spruce and aspen, the effect of drying was dependent upon the particle size of the material. For materials less than 250 μm in size, ovendrying resulted in lower calorific values than did air-drying. Drying method did not affect the heating value of the 2,000-3,350 μm material. These results indicate that the potential calorific value of wood will be lowered by comminution and ovendrying. The calorific value of ovendried finely ground spruce was approximately 4.3 percent lower than that of air-dried 2,000-3,350 μm material. As indicated by the comparison of mean values (Table 9), there was no statistical difference between the ovendried fine spruce and air-dried fine aspen. Such possible overlapping illustrates the necessity of utilizing standardized preparatory techniques in oxygen bomb calorimetry.

The reductions in calorific values may be partially attributed to a loss of volatile extractives during comminution and ovendrying. This phenomenon is not possible to fully evaluate since the moisture content used to adjust calorific values of air-dried material to a dry basis may have been erroneous due to loss of volatiles during ovendrying. However, examination of calorific values which were not adjusted for moisture still revealed distinct differences. Average unadjusted calorific value of the fine spruce which was ovendried and stored over the salt solution was 18.34 MJ/kg, compared to 18.74 MJ/kg for fine material which had not been ovendried.

According to Browning (1967), the volatile content of wood will be reduced by grinding, sieving, and drying. The author suggested that determination of volatiles should be conducted with a minimum exposure of the wood to the atmosphere. It appears that the commonly utilized techniques of comminution and ovendrying in oxygen bomb calorimetry may result in underestimates of true calorific value, especially in species with a high volatile content.

As was observed in the particle size experiment, pelletization was found to have an effect upon measured calorific value.

The calorific value of fine ovendried pelletized spruce was 20.13 MJ/kg, or about 2.3 percent higher than respective non-pelletized material. Although this suggests incomplete combustion of the loosely packed materials, inspection of the bomb contents did not reveal differences in reaction behaviour. The calorific values of pelletized fine particles of spruce however were lower than those of the non-pelletized 2,000- 3,350 μm air-dried and ovendried materials.

Due to the small number of samples tested, the effect of pelletization cannot be fully elucidated in this study. Differences in pelletized sample density may effect the rate and products of combustion and result in variations in measured calorific values. Pelletized density in turn will be dependent upon such factors as pelletizing pressure, particle size, species, and moisture content.

3.3 Radial Position Experiment

3.3.1 Specific Gravity of Hybrid Poplar Wood

Figure 5 illustrates the variation in hybrid poplar wood specific gravity with growth ring number and postion in growth increment. Analysis of variance (Table A7) showed that position in ring (earlywood vs. latewood) had a significant effect upon specific gravity. The average specific gravity of the earlywood was 0.29, compared to 0.37 for latewood. The higher specific gravity of the latewood reflects the presence of thicker walled wood

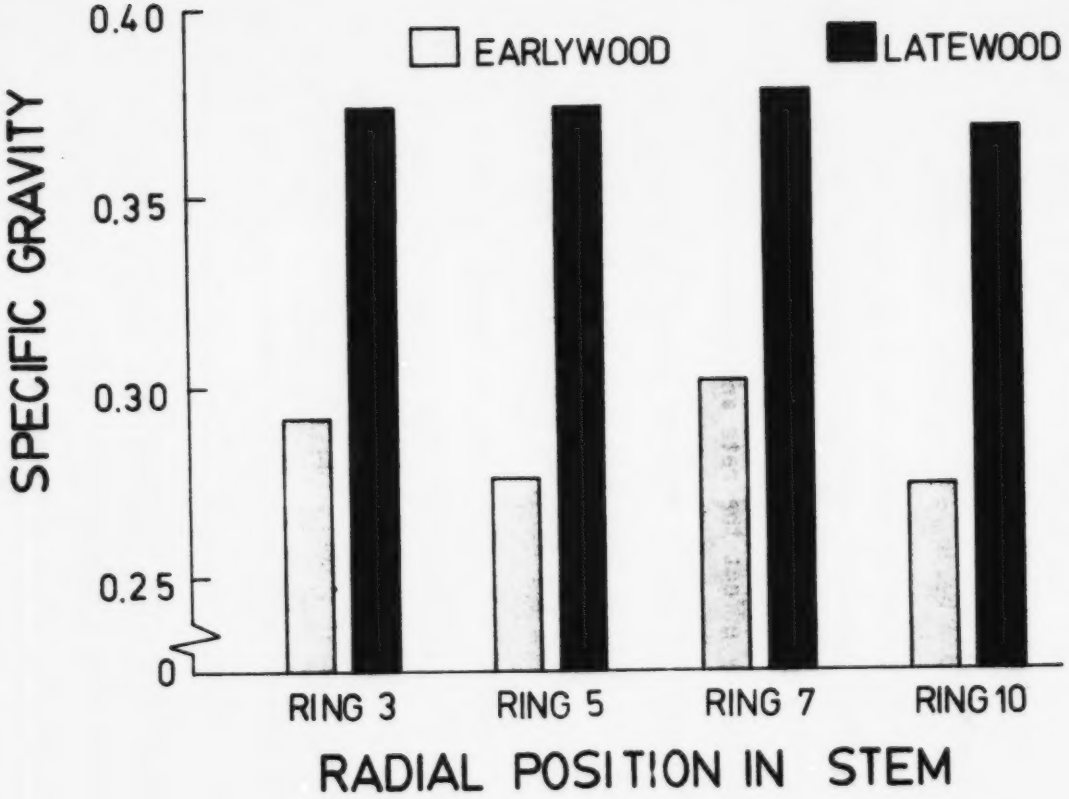

Figure 5. Specific gravity of earlywood and latewood of hybrid poplar clone I-262 (ring number represents number from pith).

cells. There was no discernible effect of growth ring number upon specific gravity. Average specific gravities of rings 3,5,7 and 10 were 0.33, 0.32, 0.34, and 0.32, respectively. Previous studies of rapidly grown hybrid poplar (Sastry and Anderson 1980, Bendtsen et al 1981) have not revealed distinct trends in specific gravity with annual ring position. For native hardwoods, including cottonwood, specific gravity has been shown to increase with increasing ring number from pith (Bendtsen et al 1981, Foster and Thor 1979, Taylor 1979, Land and Lee, 1981).

3.3.2 Constituent Analysis of Hybrid Poplar Wood

Table 10 summarizes the chemical constituents of the heartwood and sapwood of hybrid poplar. Both lignin and extractive concentrations were higher in the heartwood (ring 4) than in the sapwood (ring 9) portion of the material. Lignin content was 22.5 percent for the sapwood compared to 23.5 percent for the heartwood. It has been shown (Ritter 1953, Erickson and Arima 1974) that Klason lignin is generally 1 to 2 percent higher in the hearwood than in the sapwood of both hardwoods and softwoods. The mean lignin contents of the hybrid poplar examined in this study compare to values presented by Marton et al (1968), which ranged from 20.4 to 26.8 percent for three different clones.

Table 10. Constituent and extractive contents of sapwood and heartwood of hybrid poplar clone I-262*

Species	Lignin	Holocellulose	α-Cellulose	Extractives
	%			
Sapwood	22.5	78.3	42.3	4.4
Heartwood	23.5	78.0	42.3	5.2

*Values based upon ovendry unextracted wood. Lignin and α-cellulose are average of 2 observations while holocellulose and extractive values are based upon 1 observation. Sapwood material was sampled from ring 9 (from pith) while heartwood was from ring 4.

The holocellulose content (corrected for residual lignin of 2.7%) was approximately 78 percent for both the heartwood and sapwood. This value is comparable to the holocellulose content of trembling aspen. The α-cellulose content of the hybrid poplar was about 42 percent for both heartwood and sapwood which was approximately 4 percent lower than for trembling aspen. This indicates that the hybrid poplar had a higher pentosan content than did the trembling aspen.

Summation of lignin, holocellulose, and extractive contents resulted in values of 105.2 and 106.8 percent for sapwood and heartwood, respectively. According to Browning (1967), summations of 95-102 percent are common in practice. Overlapping, as was observed for the hybrid poplar, may have been caused by the presence of protein or polysaccharide degradation products in the isolated lignin and by residuals of ash in the holocellulose and lignin preparations. Although attempted, the samples of isolated constituents were too small to permit accurate determination of ash content.

3.3.3 Elemental Analysis of Hybrid Poplar Wood

Carbon, hydrogen, nitrogen and ash contents of the earlywood and latewood of annual rings 3,5,7 and 10 are shown in Table 11. Analysis of variance summaries for these components are listed in Tables A8-A11.

Carbon content was significantly affected (1% level) by position within the growth ring. The mean earlywood carbon content was 49.33 percent compared to 48.91 percent for latewood. This phenomenon will be discussed in more detail in a later section. Comparison of mean values (Table 11) showed that the highest carbon value was measured for the earlywood of ring 5 (49.91%). The mean carbon content of the hybrid poplar wood was 49.12 percent.

Hydrogen content was not affected by either position in the ring or distance from the pith. The average hydrogen value was 5.94 percent.

The mean wood nitrogen content was 0.53 percent. Distance from pith was observed to have a significant effect (1% level) upon nitrogen content. Comparison of mean values (Table 11) indicated that ring 10 had the highest nitrogen content (0.58%) of all rings examined.

Table 11. Elemental analysis of 4 wood growth rings and inner and outer bark of hybrid poplar clone I-262*

Sample	Carbon	Hydrogen	Nitrogen	Oxygen	Ash
	%				
Ring 3 E**	48.71b	5.83b	0.56ab	43.54	1.36a
L	49.24ab	5.99a	0.50bc	43.09	1.18a
Ring 5 E	49.91a	5.98ab	0.53ab	42.67	0.91b
L	48.86b	5.91ab	0.54ab	43.74	0.95b
Ring 7 E	49.35ab	5.92ab	0.50bc	43.53	0.70c
L	48.62b	5.96ab	0.43c	44.37	0.62cd
Ring 10 E	49.35ab	5.96ab	0.57ab	43.67	0.45d
L	48.91b	5.99a	0.60a	44.04	0.46d
Inner bark	43.23c	5.30c	0.77d	44.15	6.55e
Outer bark	50.55d	5.42d	0.33e	40.17	3.53f

*Values based upon ovendry wood. Carbon, hydrogen, and nitrogen are average of 3 observations while ash values are average of 2 observations; oxygen determined by difference. Column means with the same letter are not significantly different at the 5 percent level.

**E = earlywood, L = latewood

Wood ash content was significantly affected (1% level) by growth ring position, decreasing with increased growth ring number from the pith. Average ash contents of rings 3,5,7 and 10 were 1.27, 0.93, 0.66 and 0.46 percent, respectively. Studies of both hardwoods and softwoods (Ritter 1953, McMillin 1969) have shown that higher levels of ash are present in heartwood than in sapwood.

Although the mean ash content of earlywood was somewhat higher than for latewood (0.85 vs.0.80%), the difference was not significant. The small number of samples tested may have prohibited definitive analysis. McMillin (1969) observed that loblolly pine earlywood had a significantly higher ash content than did latewood (0.430 vs.0.389%).

The ash contents of the hybrid poplar were substantially higher than those measured for trembling aspen and white spruce. Wood ash contents of North American hardwoods and softwoods typically range from 0.2 to 0.8 percent (Panshin and de Zeeuw 1970) while ash contents of 2 to 3 percent are not uncommon for wood of tropical species (Doat 1977). Ash contents of *Populus maximowiczii* x *trichocarpa* clone NE-38 juvenile wood were found to range from 0.47 to 0.54 percent (Holt and Murphey 1978). Bowersox et al (1979) reported ash values ranging from 0.6 to 1.2 percent for mixed wood and bark samples from 7 juvenile hybrid poplar clones. The high levels of ash

observed in hybrid poplar clone I-262 may be associated with factors such as fertilization levels, soil properties, site conditions, and clonal attributes.

Ash contents of extractive-free heartwood (ring 4) and sapwood (ring 9) were found to be 0.30 and 0.23 percent, respectively (based on ovendry unextracted wood mass). These samples were prepared for chemical analyses and were not tested for ash prior to extraction. It appears reasonable, however, to assume that their original ash contents were similar to those of the adjacent rings. On this assumption, ash contents of both heartwood and sapwood were reduced after extraction. Based upon the initial unextracted ovendry mass, ash content of the unextracted heartwood was 0.93 percent (ring 5) compared to 0.30 percent (ring 4) after extraction. This indicates that approximately 68 percent of the ash in the heartwood was removed during extraction and that about 12 percent of the extractives consisted of ash. For the sapwood, extraction removed about 50 percent of the ash which constituted approximately 5 percent of the total extractives mass. Thus when ash is included in summative analyses, some overlapping is possible whereby a component of the ash is reported in the extractives concentration. To eliminate this potential

overlapping, it is recommended that for wood and bark with high levels of inorganics, ash content be determined prior to and after extraction. Any reduction in ash level due to extraction should then be deducted from the apparent extractive content. The nature of the combination of ash in wood is largely unknown (Browning 1967) and further research appears needed to fully clarify this phenomenon.

3.3.4 Elemental Analysis of Hybrid Poplar Bark

Elemental analysis revealed distinct differences between the chemical composition of the inner and outer bark. Carbon, hydrogen, nitrogen, and ash levels of inner bark were all significantly different from respective outer bark values. Carbon content of the outer bark was 50.55 percent compared to 43.23 percent for the inner bark. Outer bark displayed higher hydrogen and lower nitrogen levels than did the inner component. These variations can be attributed to basic differences in constituent concentrations. Analyses of barks of various hardwoods have shown that outer bark contains more lignin and less holocellulose than does inner bark (Binotto and Murphey 1975, Sandala et al 1981).

Ash content of the inner bark was 6.55 percent compared to 3.53 percent for outer bark. The high ash levels in the hybrid poplar bark may attribute to substantial overlapping in constituent determinations.

3.3.5 Calorific Values of Hybrid Poplar Wood

Calorific values of the various hybrid poplar growth rings are listed in Table 12 and presented graphically in Figure 6. Analysis of variance for rings 3,5,7 and 10 (Table A12) showed that wood calorific value was significantly affected (1% level) by growth ring position. In general, calorific value exhibited a cyclic pattern with maximum values occurring in the middle growth rings. Comparison of average (combined earlywood and latewood) calorific values of rings 3,5,7 and 10 showed that the heating value of ring 10 (19.20 MJ/kg) was significantly lower than for the other growth rings. Ring 5 displayed the highest calorific value (19.68 MJ/kg), which was somewhat higher than that of ring 7 (19.56 MJ/kg). The calorific value of ring 3 (19.53 MJ/kg was comparable to that of ring 7 but significantly lower than the heating value of ring 5. This may be attributed to the high ash content of ring 3. Although significant, the difference between average minimum and maximum growth ring calorific values amounted to only less than 3 percent. Comparisons of individual earlywood and latewood mean values are listed in Table 12.

Table 12. Calorific values of 5 wood growth rings and inner and outer bark of hybrid poplar clone I-262*

Sample	Calorific value (MJ/kg)			
	Experimental		Theoretical according to IGT equation [1]	Error (%)
	Average	Range		
Ring 1 E&L**	19.66a	19.56 - 19.74	-	-
Ring 3 E	19.66a	19.57 - 19.77	19.08	-2.95
L	19.39bc	19.18 - 19.53	19.54	+0.74
Ring 5 E	19.71a	19.59 - 19.79	19.80	+0.48
L	19.66a	19.61 - 19.72	19.22	-2.25
Ring 7 E	19.60a	19.48 - 19.72	19.44	-0.81
L	19.52ab	19.45 - 19.61	19.14	-1.95
Ring 10 E	19.19d	19.16 - 19.20	19.47	+1.50
L	19.20cd	19.07 - 19.30	19.31	+0.56
Inner bark	16.62e	16.47 - 16.74	16.33	-1.75
Outer bark	20.14f	20.04 - 20.22	19.56	-2.90

* Values based upon ovendry wood. Experimental averages are based upon 3 observations. Experimental mean values with the same letter are not significantly different at the 5 percent level.

** E = earlywood, L = latewood

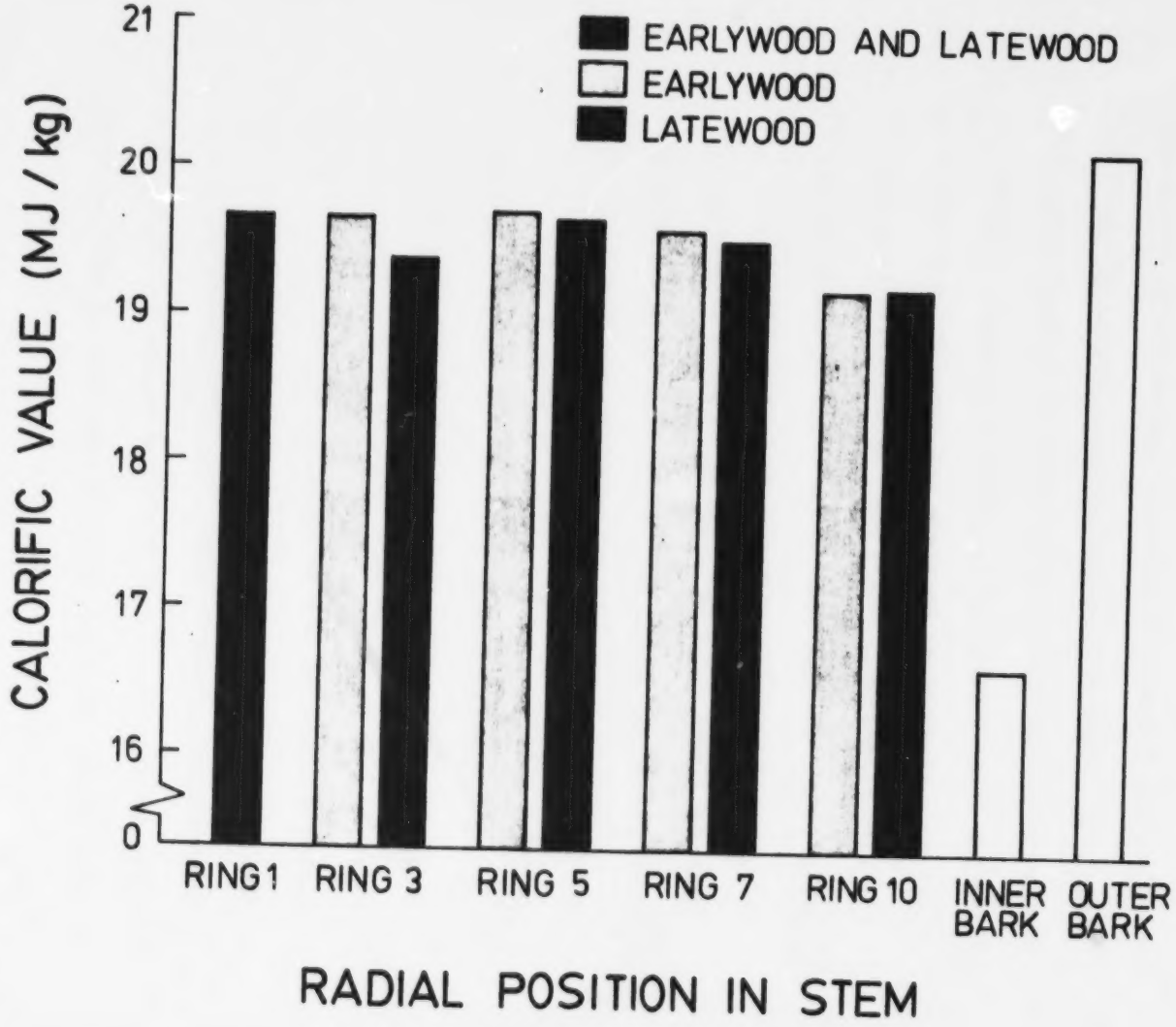

Figure 6. Calorific values of wood and bark of hybrid poplar clone I-262 (ring number represents number from pith).

The higher calorific values observed in the inner rings can be attributed to larger amounts of lignin and extractives, although elemental analysis did not detect a significant change in carbon or hydrogen with distance from pith. However, the maximum calorific value was measured on the ring component with the highest carbon content (ring 5 earlywood). Table 12 presents theoretical wood calorific values obtained using the IGT and the ultimate elemental analysis data of Table 11. For ring 5 earlywood, the IGT equation resulted in a theoretical calorific value of 19.80 MJ/kg, or 0.48 percent higher than the experimental value of 19.71 MJ/kg. As with the experimental data, the theoretical value was also a group maximum. However, the IGT equation tended to underestimate the heating values of rings 3 and 7 and overestimate those of ring 10. In the case of ring 3 earlywood, the equation underestimated the experimental value by 2.95 percent. Thus the use of the elemental analysis data with the IGT equation did not fully detect the cyclic radial pattern which was exhibited by the experimental calorific data. The IGT equation and elemental data resulted in an average hybrid poplar heating value of 19.38 MJ/kg, compared with the experimental mean value of 19.49 MJ/kg.

The radial variability of wood calorific value has not been fully evaluated. Howard (1973) found that the calorific value of southern pine wood was positively correlated to extractive content, which decreased with increasing distance from the pith. Similarly, Doat (1977) evaluated 111 tropical species and found that calorific value was positively correlated to lignin plus alcohol-benzene soluble extractives. Calorific value of trembling aspen was observed to increase slightly radially from pith to bark (Sastry and Anderson 1980). The authors observed a cyclic pattern for hybrid poplar clone DN-30 in which the lowest calorific values were measured for the inner and outer growth rings. The differences however, appeared substantially larger than those observed for hybrid poplar clone I-262 in this study.

The average calorific value of the hybrid poplar earlywood was found to be slightly higher than that of latewood (19.54 vs.19.45 MJ/kg). Howard (1973) observed that the calorific value of southern pine earlywood was somewhat greater than for latewood, although the difference was not significant. Intra-incremental studies of softwoods have shown that earlywood generally contains a few percent more lignin than does latewood (Wilson and Wellwood 1965). Proportionally, earlywood cells contain more middle lamella and less secondary wall than do latewood cells. The secondary wall contains the majority of the cell holocellulose

whereas the middle lamella contains a high concentration of lignin. The observed within-ring variation of hybrid poplar specific gravity would presumably be associated with a gradient in the constituent composition. The presence of such a gradient is supported by the elemental analysis data which showed that earlywood contained more carbon that did latewood. Using the elemental data and the IGT equation, the average theoretical heating value for earlywood was 19.45 MJ/kg, or 0.46 percent lower than the experimental mean value of 19.54 MJ/kg. For latewood, the theoretical value was 19.30 MJ/kg, or 0.74 percent lower than the experimental value of 19.45 MJ/kg.

3.3.6 Calorific Values of Hybrid Poplar Bark

The calorific value of inner bark was 16.62 MJ/kg, compared to 20.14 MJ/kg for outer bark (Fig. 6, Table 12). As discussed in section 3.3.4, the outer bark contained more carbon and hydrogen and less nitrogen and ash than did the inner bark. Utilizing the IGT equation and the elemental composition data, theoretical calorific values of the inner and outer bark were calculated as 16.33 and 19.56 MJ/kg, respectively.

For hybrid poplar clone DN-30, Sastry and Anderson (1980) found that the heating value of inner bark was about 5 to 10 percent lower than that of the outer bark. Sandala et al (1981) reported that calorific values for inner bark of 3 native hardwoods were between 14 and 25 percent lower than for corresponding outer bark values.

Inappropriate sampling and preparation techniques may lead to variations in the calorific analysis of bark. According to Binotto and Murphey (1975), inner and outer bark are different populations and should be separated prior to chemical analysis. Ottone and Baldwin (1981) have shown that inner and outer bark will fractionate into different particle size classes during grinding. Sieving of whole-bark for calorific testing may consequently produce a sample which is not representative of the original material. Furthermore, whole-bark calorific values may vary significantly with stem diameter, since the ratio of outer to inner bark increases with increased tree diameter.

4. SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

Recent research conducted at the University of Toronto has shown that a decrease in particle size will result in an increase in the gross heating value of juvenile hybrid poplar wood and/or bark. These results indicated that it may be economically advantageous to reduce wood to a fine particle size for utilization in wood energy conversion systems. Since the initial University of Toronto tests were conducted on heterogeneous materials, a study was contracted to Forintek Canada Corp. to fully evaluate the effects of particle size and other possible factors on the calorific value of wood.

The major objective of this research was to examine the effects of (a) particle size, (b) drying method, and (c) radial position in stem upon the calorific value of wood. Oxygen bomb calorimetry was conducted on: (a) 6 particle sizes (2,000-3,350, 250-425, 150-250, 75-150, 38-75 and ‹38 μm) of trembling aspen and white spruce wood obtained from kiln-dried lumber,(b) 2 particle sizes (2,000-3,350 and ‹250 μm) of white spruce and trembling aspen which had been either air-dried or ovendried from the green state, and on (c) inner and outer bark and earlywood and latewood samples from various growth rings of hybrid poplar clone I-262. In (c), only 1 particle size (‹250 μm) was utilized for calorific testing. Calorific values were determined at a moisture content of 6 to 7 percent (wet basis) and then adjusted to a dry basis.

Lignin, holocellulose, and α-cellulose contents were determined on ovendried 180-250 μm wood from the aspen, spruce, and hybrid poplar. Selected wood and bark samples were analyzed for carbon, hydrogen, nitrogen, and ash concentrations. Experimentally determined calorific data were compared to theoretical values calculated using the IGT correlation equation. The results of this study are summarized as follows.

(a) Particle size was found to have a significant effect upon the calorific values of both spruce and aspen (initially kiln-dried). The calorific values of the coarse (2,000-3,350 μm) and fine (38-75 and ‹38 μm) materials were generally higher than those of the intermediate particle sizes. Although significant, the differences between average minimum and maximum calorific values were less than 3 percent. Spruce was found to have both a higher lignin content and heating value than the aspen. The general relationship between particle size and calorific value was dissimilar to the pattern observed by the University of Toronto for juvenile hybrid poplar.

(b) Comminution followed by ovendrying was found to lower the calorific values of the spruce and aspen (which had been obtained green). The calorific value (dry basis) of ovendried ‹250 μm spruce was 4.3 percent lower than that of air-dried 2,000-3,350 μm material.

(c) Calorific value of hybrid poplar wood exhibited a cyclic pattern with maximum values occurring in the middle growth rings. Although significant, the difference between minimum and maximum growth ring heating value was less than 3 percent. The lowest calorific value was measured for the outermost growth ring. Correspondingly, the hybrid poplar sapwood had slighly lower lignin and extractive contents than the heartwood. The average calorific value of earlywood was about 0.5 percent higher than that of latewood, although the difference was not significant. Wood ash content decreased with increasing growth ring number from pith. The heating value of inner bark was approximately 17.5 percent lower than that of outer bark. Theoretical heats of combustion calculated using the IGT equation were usually within 3 percent of the experimentally determined values.

In conclusion, this study did not find evidence to suggest an overall energy gain by comminuting clear wood samples to a fine particle size. However, since calorific value was found to vary somewhat with particle size, it is recommended that additional work be conducted on heterogeneous materials, such as hybrid poplar, which may be subject to fractionation into particle sizes of differing chemical composition during comminution. This phenomenom may have attributed to the variations in the calorific values of hybrid poplar wood and/or bark reported by the University of Toronto.

Since this current study indicated that comminution, ovendrying, radial position in stem, and pelletization may significantly affect calorific value, it is recommended that additional research be conducted utilizing wood and bark of various species in order to determine the most appropriate preparatory techniques for adiabatic oxygen bomb calorimetry. This would reduce potential errors and permit direct comparison of calorific values, which are, currently commonly presented with little or no reference to sample preparation.

It is emphasized that the conclusions and recommendations of this work apply strictly to combustion data as determined on small, clear samples by adiabatic oxygen bomb calorimetry. For example, this study does not intimate that the thermal efficiencies of commercial combustion units will not be increased through feedstock comminution. Similarly, the calorific values of small, clear wood samples will presumably be significantly different than the heating values of the heterogeneous forest biomass mixtures which will likely be utilized as an energy source in the future. To overcome these limitations, it is recommended that a large-scale calorimeter be developed to evaluate such factors as composition, moisture content, particle size, and particle configuration of commercially representative types and quantities of biomass materials.

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6. APPENDIX - Statistical Data

Table A1. Analysis of variance - carbon content of 250-425 and 38-75 µm trembling aspen and white spruce, particle size experiment.

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	2	0.9978	0.4989
Particle size (P)	1	0.0979	0.0979
Species (S)	1	1.9441	1.9441
P x S	1	0.0161	0.0161
Error	6	1.8219	0.3036

Table A2. Analysis of variance - hydrogen content of 250-425 and 38-75 µm trembling aspen and white spruce, particle size experiment.

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	2	0.0513	0.0257
Particle size (P)	1	0.0125	0.0125
Species (S)	1	0.0000	0.0000
P x S	1	0.0091	0.0091
Error	6	0.0671	0.0112

Table A3. Analysis of variance - nitrogen content of 250-425 and 38-75 µm trembling aspen and white spruce, particle size experiment.

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	2	0.0114	0.0057
Particle size (P)	1	0.0003	0.0003
Species (S)	1	0.0217	0.0217*
P x S	1	0.0047	0.0047
Error	6	0.0063	0.0010

*Significant at the 1 percent level.

Table A4. Analysis of variance - trembling aspen and white spruce calorific values after standard and batch comminution, particle size experiment.

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	2	0.0418	0.0209
Particle size (P)	5	1.5030	0.3010*
Species (S)	1	3.4055	3.4055*
Grinding method (M)	1	0.0175	0.0175
P x S	5	0.0954	0.0191
P x M	5	0.2931	0.0586*
S x M	1	0.1029	0.1029*
P x S x M	5	0.3619	0.0724*
Error	46	0.5687	0.0124

*Significant at the 1 percent level.

Table A5. Analysis of variance-sucrose calorific values, particle size experiment

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	2	0.0059	0.0029
Treatments	4	0.0524	0.0131
Error	8	0.0453	0.0057

Table A6. Analysis of variance - trembling aspen and white spruce calorific values, drying experiment

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	2	0.0102	0.0051
Species (S)	1	3.7257	3.7257*
Particle size (P)	1	0.4851	0.4851*
Drying method (D)	1	0.2748	0.2748*
S x P	1	0.5545	0.5545*
S x D	1	0.0440	0.0440
P x D	1	0.1356	0.1356*
S x P x D	1	0.0084	0.0084
Error	14	0.0956	0.0068

* Significant at the 1 percent level.

Table A7. Analysis of variance - specific gravity of earlywood and latewood of rings 3, 5, 7, and 10, hybrid poplar clone I-262, radial position experiment.

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	2	0.0001	0.0001
Position within ring (W)	1	0.0464	0.0464*
Ring number from pith (R)	3	0.0013	0.0004
W x R	3	0.0005	0.0002
Error	14	0.0015	0.0001

*Significant at the 1 percent level.

Table A8. Analysis of variance - carbon content of earlywood and latewood of rings 3, 5, 7, and 10, hybrid poplar clone I-262, radial position experiment

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	2	0.0453	0.0226
Position within ring (W)	1	1.0757	1.0757*
Ring number from pith (R)	3	0.6630	0.2210
W x R	3	2.0811	0.6937
Error	14	2.2553	0.1611

*Significant at the 1 percent level.

Table A9. Analysis of variance - hydrogen content of earlywood and latewood of rings 3, 5, 7, and 10, hybrid poplar clone I-262, radial position experiment.

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	2	0.0196	0.0093
Position within ring (W)	1	0.0099	0.0099
Ring number from pith (R)	3	0.0150	0.0050
W x R	3	0.0406	0.0135
Error	14	0.0921	0.0066

Table A10. Analysis of variance - nitrogen content of earlywood and latewood of rings 3, 5, 7, and 10, hybrid poplar clone I-262, radial position experiment.

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	2	0.0116	0.0058
Position within ring (W)	1	0.0034	0.0034
Ring number from pith (R)	3	0.0430	0.0143*
W x R	3	0.0113	0.0038
Error	14	0.0318	0.0023

*Significant at the 1 percent level.

Table A11. Analysis of variance - ash content of earlywood and latewood of rings 3, 5, 7, and 10, hybrid poplar clone I-262, radial position experiment.

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	1	0.0023	0.0023
Position within ring (W)	1	0.0105	0.0105
Ring number from pith (R)	3	1.4905	0.4968*
W x R	3	0.0282	0.0094
Error	7	0.0381	0.0054

*Significant at the 1 percent level.

Table A12. Analysis of variance - calorific values of earlywood and latewood of rings 3, 5, 7, and 10, hybrid poplar clone I-262, radial position experiment.

Source of variation	Degrees of freedom	Sum of squares	Mean square
Replications	2	0.0480	0.0240
Position within ring (W)	1	0.0500	0.0500
Ring number from pith (R)	3	0.7833	0.2611*
W x R	3	0.0680	0.0227
Error	14	0.1430	0.0102

*Significant at the 1 percent level.